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The mechanical behavior of the material-tissue and material-material interface in dental reconstructions

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Contents

Abstract

In dental and craniofacial sciences, frequently the goal is to replace lost or damaged natural tissue with synthetic materials. For ideal function, these replacement materials must strongly bond to the existing tissue, but they also must form a hermetic seal that eliminates the passage of microorganisms and fluids that would lead to further tissue destruction or weakening of the interface or the individual materials, compromising the final outcome. Therefore, the study of interfaces is crucial, and the manner in which they can be tested to predict the likelihood of success is of great interest to the field.

Because a variety of materials and material combinations are used for the repair or replacement of oral and craniofacial tissues, numerous types of material interfaces exist. A complete discussion of this important topic requires an examination of all of them. In this review article, the three different types of interfaces are treated separately. First, the interface between the tooth tissue and restorative material is explored, specifically by considering resin-based materials such as dental adhesives and composite, and the manner in which they interact with dentin and enamel. Second, the interaction between these same resin-based materials and other structures, such as oxide ceramic dental crowns, are explored, because these tooth replacement materials are typically fixed to the remaining tooth structure through the use of resin-based adhesives and cements, or repaired intraorally with similar materials. Finally, the interface between different synthetic materials, such as metals and ceramics, with dental porcelain used as an esthetic veneering material is addressed.

Keywords: Interface, dental adhesion, bond strength test, resin cement, dental ceramics, zirconia, veneering ceramics

1. The Resin Adhesive and Tooth Interface

1.1 Mechanical properties of enamel and dentin

Tooth structure is composed predominantly of hydroxyapatite, a calcium-phosphate crystalline mineral (Figure 1). The enamel is almost totally mineralized, incorporating only a very small amount of organic matrix, being mainly protein (3 vol%), and water (12 vol%) [1]. The crystals are arranged in long rods having a very well defined radial anisotropy. Dentin is composed of approximately 47 vol% mineral, and containing 33 vol% of protein, mainly collagen with other non-collagenous proteins, and about 20 vol% water [1]. Dentin is arranged in a tubular structure, with a lower density of tubules near the dentin-enamel junction, and a greater tubular density, and subsequently lower mineral density, near the dental pulp. Therefore, the properties of dentin vary based on location within the tooth.

Enamel, typical of many ceramics, is a very brittle material, possessing high elastic modulus (~70-110 GPa) but relatively low tensile strength (10-70 MPa) and fracture toughness (0.7-2.1 $MPa \cdot m^{1/2}$) [2]. Dentin, in contrast, is of much lower elastic modulus (~20 GPa) than enamel, but has higher tensile strength (60-100 MPa) and is more fracture resistant (1.5-2.1 $MPa.m^{1/2}$), all due to its higher organic content [2]. Enamel is also about 5-6 times harder than dentin. The most important aspect of the enamel-dentin complex is the interface, or transition region (Figure 2), between the two, called the dentin-enamel junction (DEJ), which is estimated to be anywhere from 10-100 μm thick [3], though Gallagher et al. [4] used a combination of nano-indentation and Raman micro-spectroscopy to estimate the thickness as less than 10 μm.

There are many views as to how the DEJ provides enhanced toughening of the tooth, including the mismatch in elastic modulus between the dentin and enamel that arrests cracks [3] and crack bridging and localized microcracking within the enamel as the junction is approached [5]. The ultimate outcome is that there is significant energy dissipation at the interface, which often allows cracks to reach, but not

extend into the dentin. In addition, there is strong adhesion between the enamel and dentin at the interface, with an apparent increase in fracture toughness as the DEJ is approached [3]. Thus, the DEJ provides a dramatic advantage over the individual tooth components that accounts for the fact that a very high proportion of teeth in humans have cracks, but they continue to function for many years, despite the fact that the outer enamel structure is compromised [6, 7].

1.2 Mechanical properties of the adhesive interface

In modern dentistry, it is most typical to create bonded interfaces with enamel and dentin through resin-based adhesives and composites, both being composed of polymer matrices, and the latter especially being reinforced by inorganic particulates coupled to the polymer with a silane coupling molecule, thus securing that interface within the material. Resins are capable of flowing into micro- and macroscopic irregularities on the surface of the enamel or dentin and "locking" into the tooth structure once the resin becomes hardened. This adhesive force is predominantly a result of mechanical forces, but also derives from secondary bonding, with a potential for chemical interactions for certain adhesive systems.

A comparison of several adhesive resins that have been tested in many studies over the years shows average adhesive bond strengths to dentine from approximately 22 to 54 MPa using the microtensile mode, with an actual range of 6-75 MPa, and an average of 12 to 26 MPa with the shear test method, with an actual range of 6-39 MPa [8]. This data suggests that the adhesive strength of the interface may be approaching, or even exceeding, the tensile strengths of the dentin and enamel, respectively.

The multilayer interface that forms between the resin and dentin may be more appropriately termed an 'interphase'. This region is composed of tooth structure and resin, and has been called the "hybrid-layer" due to its dual composition. The layer is created by acid treatment of the dentin to

demineralize and expose the collagen network, and subsequently impregnating the collagen with monomers that polymerize into a cross-linked polymer matrix surrounding the collagen fibrils. The layer may be fractions of a micrometer, as is typical with self-etching adhesives using milder organic acids, and up to several micrometers thick, as is more typical for etch-and-rinse type adhesives employing stronger acids, though the strength of the interface is not dependent upon thickness (Figure 3). This resin penetration process is usually incomplete, leaving porosities on the micro- and nano-scale within the hybrid layer that weaken it [9].

The mechanical properties of the materials across this resin-dentin interface have been investigated by many, including Van Meerbeek et al. [10] who used nanoindentation of permanent teeth to study the hardness and elastic modulus and showed an expected transition from the higher modulus dentin through the hybrid layer of lower modulus to the unfilled resin adhesive of even lower modulus. Hardness followed a similar pattern. They suggested that this "elastic layer" may provide deformation that actually enhances the adhesive to remain bonded to the tooth structure during the polymerization shrinkage of the composite restorative, especially if the adhesive layer above the hybrid layer is thick. Hosoya and Marshall [11] and Hosoya [12] measured hardness across the interface of resin with sound and carious primary dentin with nanoindentation. They showed different results than with permanent dentin in that the hardness and modulus were not significantly reduced within the interface as compared with sound dentin below the interface, and were possibly even higher when compared to carious dentin. Ryou et al. [13] used nano-DMA (dynamic mechanical analysis) to show that the storage modulus of the hybrid layer was heterogeneous due to the different properties of the resin tags and the resin-reinforced collagen areas that compose the interface. The resin-collagen interphase had slightly higher modulus than the resin itself due to the greater stiffness of the collagen, but the properties of both components, as expected, were highly dependent upon hydration state. They also verified that this layer of resin-

infiltrated collagen was extremely viscoelastic, and thus loading rate would have a significant effect on its properties, during function as well as during mechanical testing.

1.3 Interfacial testing methods

1.3.1 Tensile and shear bond strength tests

Historically, the predominant testing mode for tooth-resin interfaces was the tensile or the shear bond method, and more recently the microtensile bond method. It is likely that the purest method for testing the bond between the two materials is uniaxial tension of specimens of a size that are indicative of dental restorations. In this case, the material, in the form of a cylinder, is cured directly on a dentin or enamel surface, and then somehow gripped and pulled up off the surface. Failure stress is calculated as the force at fracture divided by the cross-sectional area, based on the assumption that the stresses are uniformly distributed within the joint area. Uniaxial tensile testing relies on critical alignment, gripping and loading procedures that often are neglected or compromised, resulting in failures that do not provide an accurate indication of the true interfacial properties. For many, the shear test method provided a simpler approach. However, with either method, non-uniform stress distributions are typical at either the edges of the interface or in the center of the joint due to specimen production limitations and geometries, and these stress concentrations can significantly affect testing outcomes. It is common, and predictable, that failures originate from defects and critical flaws in the adhesive resin material near the interface, typically at the fillet formed during the joining/curing process, rather than directly at the interfacial joint [14].

Studies have also verified that cohesive failure of the components, typically the tooth structure, occurs due to these stress concentrations [15]. This is due to the more brittle nature of dentin and enamel, and the fact that in shear tests, the composite upon which the load is applied is mostly in

compression while the tooth structure is predominantly in tension, making failure via crack propagation directed within the tooth structure more likely. If the two materials at the interface were more closely matched in terms of mechanical properties, cracking along the interface would be expected.

Using finite element analysis, Van Noort et al. [16] examined these variables, as well as the manner in which the properties of the dental composite affect stress distributions. In shear test modes, they found that peak interfacial stresses could be 65% higher with composites of higher modulus. They concluded that though the actual adhesive strength of the interface might be the same, composites with greater modulus could show higher interfacial stress values simply due to the influence of their own mechanical properties, and the difference between the properties of the composite and the tooth. This outcome helped to explain a wide variability in the results of composite bond strength to tooth structure. Similarly, in tensile testing, the thickness of the materials on either side of the test joint influence stress distribution, with thinner sections causing greater stresses at the center of the joint as opposed to at the edges [17].

Another significant issue with shear stress tests is the location of the applied load to the actual tooth-resin interface. There are large stress concentrations in the material at the site of load application, the magnitude of which depends on the contact area between the loading device and the composite [18]. Point source load applications understandably produce the highest stresses. In any case, the stresses are highest within the interface closest to the loading site, and diminish and become more uniform within the bulk of the joint. For this reason, some have suggested using a wire loop, as compared to a knife-edge, to engage more area of the composite cylinder during loading to minimize the stress concentrations. In either situation, tensile stresses become more prevalent than shear as the loading site moves further away from the interface [17]. In any case, these analyses show that the method of calculating bond strength by dividing failure load by cross-sectional area severely underestimates the true failure stress in the bonded interface [19].

1.3.2 Microtensile bond strength tests

In an attempt to eliminate the failure of dentin during bond tests, and to be able to obtain multiple test specimens from various parts of a single tooth, the micro-tensile bond test was introduced by Sano et al. [20]. With this method, composite can be bonded either to a flat dentin or enamel surface, or within a cavity preparation in a whole tooth, and individual stick-shaped specimens, approximately 1 mm square, are cut providing a bonded interface that can be tested in tension. The sticks are tested with a square cross-section, or as originally designed, further shaped into a dumbbell to ensure failure at the interface. Typically, due to the smaller surface area being tested, bond strengths using the microtensile mode are higher than those obtained from macroscale tensile or shear tests, but the methods are highly correlated and tend to rank materials in the same order [9].

The quality and stability of the resin-dentin interface has been studied exhaustively. A clinical study has verified that the bond strength declines with time in the mouth [21]. The lack of stability of this interface is associated with a degradation of the exposed collagen and plasticizing of the polymer resin occurring with time, leading to enhanced internal porosity and a weakened structure. This has been verified both in vivo [22] and in vitro [23].

The utility and appropriateness of interfacial strength testing using the microtensile test has been the subject of numerous papers [24,25]. Some have suggested that there is only very limited correlation of such tests with actual clinical performance of materials [26]. Recent reviews suggest that “simple” tests of interfacial bond strength via shear or microtensile methods, in general, are plagued by high levels of data scatter, in large part due to the fact that many failures are not truly interfacial, though they are considered as such in the data analysis [27]. Often fractographic analysis of debonded interfaces is performed with optical microscopy with insufficient resolution to identify the true site of fracture, leading to the identification of so called “mixed fractures” in which the failure appears to have occurred

within the substrates as well as at the interface. Thus, the true bond strength cannot be determined [19]. These uncertainties cast considerable doubt upon the extent to which these tests then can actually predict the interfacial adhesion of the clinical situation, even if standardized test methods are employed. For these reasons, many have argued for adopting an alternative approach to bond strength testing involving fracture mechanics [15,18, 28-31].

1.3.3 Fracture mechanics

The first test of the fracture toughness of the interface between resin and tooth structure was conducted by DeGroot et al. [32] for dental enamel and composite using the single edge notched beam method. They showed that the obtained value for the critical stress intensity, K_{Ic}, ranged from 0.84-1.02 $MPa \cdot m^{1/2}$ in the case of interfacial failure, being greater for the more heavily filled, stiffer composite. As noted above, higher bond strength values have been shown for stiffer composite using more conventional test methods. This work was followed by Tam and Pilliar [28] using the chevron notch method for composite bonded to dentin. They reported fracture toughness values of 0.2-0.7 $MPa \cdot m^{1/2}$ $MPa \cdot m^{1/2}$ depending upon the type of adhesive. They also measured the value for the enamel bonded to composite interface as 1.11 $MPa \cdot m^{1/2}$, which is fairly consistent with the results of [32]. Tam and Pilliar [30] further assessed the microstructure of the fracture surfaces using SEM and x-ray microanalysis, which helped to explain the differences in toughness values for the different adhesives based on extent of resin penetration and site of failure.

Many others have measured the fracture toughness of the interfacial bond [34-36]. Armstrong et al. [35] identified the weakest interfaces in the joint being at the top of the hybrid layer and adhesive interface and at the bottom of the hybrid layer and dentin interface when tested with chevron notched short bars. They also confirmed that when testing with the microtensile method, failures were typically within the substrates near the interface. A study by DeMunck et al. [37] compared the results of

microtensile bond strength tests with the chevron notch beam (CNB) method for fracture toughness, and showed that although there was a reasonable correlation between the two methods for six dentin adhesives, there was significantly less scatter in the results for the CNB method. Further, most failures were interfacial for the CNB method, where most were not for the microtensile method. One may conclude that fracture toughness may serve as a more accurate and precise mode for testing the interfacial quality of the resin-tooth structure bond, though it may be more laborious in terms of specimen preparation and methodology, requiring a very sharp notch, or pre-crack, be placed precisely at the bonded interface.

1.3.4 Fatigue

Dong and Ruse [38] produced crack propagation across the DEJ through fatigue load cycling, and showed through scanning electron microscopy that the DEJ deflected cracks initiated in the enamel and running parallel to the plane of dentin tubules to a nearly perpendicular direction. They also measured fracture toughness using the notchless triangular prism geometry and recorded values for the complex of 1.5 $MPa \cdot m^{1/2}$, which was consistent with other studies. Drummond et al. [39] compared static shear tests to cyclic fatigue and shear punch bond tests and showed that the cyclic fatigue method provided a more conservative assessment of the true interfacial bond strength, being essentially one-half the value as that obtained in the other tests. They used a staircase approach where the cyclic stress level is increased or decreased by a defined level (i.e. like a stair step) for each subsequent test based on the results (failure or survival of a specimen to a given number of cycles) of the previous test.

Many others have used various types of fatigue tests, including bending, compression, rotary and tensile to study various aspects of the resin adhesive-tooth interface [40-45]. In the De Munck et al. [41] and Belli et al. [43] studies, they verified that the fatigue bond strength was approximately 25-70% of the static bond strength. Both showed evidence for microstructural damage accumulation during the

fatigue testing that led to the reduced joint strength. This relationship of fatigue strength to static strength is similar to what is found for the dental composites, where the fatigue strength is typically around 50% of the static strength [46,47]. The studies by Mutluay et al. [44] and Yahyazadehfar et al. [45] introduced a novel method where the beam test specimen involves two similar bonded joints, one of which fails during the test to provide a value for the fatigue strength, and the other that allows microstructure analysis of the failure mechanisms. These studies verified that the dentin-composite joint had lower fatigue strength than the two joint materials, and that viscoelastic deformation during cyclic loading caused breakdown within the interface leading to failure, possibly through enhanced hydrolytic degradation. They also showed that for the joint with enamel, micro-fractures within the enamel itself led to failure of the joint.

The results of these studies suggest that tests involving the static evaluation of the adhesive-tooth bond are likely to overestimate the true durability of the adhesive interface. It is apparent that methods incorporating fracture toughness testing or fatigue loading are likely to be more appropriate for truly testing the interfacial integrity, and possibly for correlating with clinical performance of various materials.

2. The Resin Cement and Ceramic Interface

Resin-based cements are commonly used for permanent fixation of dental ceramics and have become the material of choice in modern adhesive dentistry providing enhanced clinical longevity compared with more traditional luting methods. Similar to the progression of resin composite restorative adhesive systems, developments in materials science have provided the dental practitioner with a variety of resin-based cement materials. However, dissimilar surface chemistries, variations in surface preparation and the degradative effects of the oral environment may significantly affect the mechanical performance of the prosthesis, specifically at the resin ceramic adhesive interface.

2.1 Resin strengthening of predominantly glassy ceramics

For several decades the use of traditional luting agents such as zinc phosphate cements were considered the gold standard for cementation purposes, and have been so successful that they are still used today. However, such materials simply act as a sealant between the tooth and restoration offering only weak micromechanical retention with no chemical adhesive potential. Although the introduction of zinc polycarboxylate and glass-ionomer cements improved the chemical affinity between tooth and restoration, the low initial acidity of the mixed cement is known to extend the inherent defect population at the ceramic surface and reduce the mechanical reliability of the ceramic-lute interface [48]. For over 15 years it has been known that the clinical longevity of traditional, predominantly glassy ceramic prostheses can be significantly improved by interfacial adhesion of the ceramic restoration to tooth structure using resin-based cements [49-52]. The surface of silica-based glassy ceramics is amenable to modification by hydrofluoric acid, which increases surface area and energy, as well as wettability of the cement system [53]. Subsequent surface priming using amphiphilic silane chemistry provides efficient coupling of the inorganic glass and organic moiety of the resin cement.

Failure of the resin-ceramic joint occurs from the extension of a critical flaw within a pre-existing defect population [54-56]. If the flaw population can be modified at the ceramic surface, the probability of extending a critical defect can be significantly reduced. Previous studies have proposed various mechanisms for improving the mechanical properties of the resin-ceramic interface by resin penetration within existing surface micro-porosities [57,58] and through the creation of a hybrid layer consisting of an interpenetrating phase of ceramic surface porosity and infiltrated resin cement [59,60].

2.2 Resin cementation of zirconia polycrystalline ceramics

Unlike ceramics that contain a silica glass phase that is readily modifiable by acid-etching, high-density polycrystalline ceramics such as zirconia are much less easily prepared. Further, direct chemical adhesion of a conventional silane primer layer is more problematic as the surface of a zirconia ceramic is more stable and comparatively non-polar, which eliminates the possibility of forming silanol groups as the surface is not readily hydrolysed [61]. Consequently, either tribo-mechanical, chemical modification, or the use of an appropriate coupling agent at the ceramic surface is required.

The literature is awash with various surface pre-treatment protocols, development of primer and adhesive chemistry and characterization of their adhesive potential between resin cements for luting purposes or resin composite restorative materials for intra-oral repair. Mechanical alteration of the ceramic surface tribology can involve various methods. Airborne particle abrasion, or 'grit-blasting' using various particle types, sizes and application [62-64] is used in order to increase roughness, surface energy and create micromechanical retentive features. Due to the superior hardness of polycrystalline ceramic materials it is difficult to roughen the surface to a similar degree to that of traditional glassy ceramics. However, previous reports suggest that adhesion to grit-blasted polycrystalline surfaces is improved, presumably by surface decontamination, increased roughness (albeit to a limited extent) and improved wettability of the resin cement material [65,66]. Laboratory findings also suggest that grit-

blasting can increase load-to-failure strengths under bi-axial flexure [67,68] or fatigue strengthening [69] as a result of the well-known phase transformation within the metastable crystal lattice of ytrria-stabilised zirconia, and subsequent volume change that may induce an increased compressive strength at the ceramic surface. On the other hand, previous researchers have suggested that surface abrasion may induce critical surface flaws that reduce mechanical properties and potentially affect the longevity of zirconia crowns [70]. Such effects are largely dictated by numerous abrasion factors; particle size/type [71], nozzle distance/orientation, duration [72] and application pressure [73]. Even if phase transformation provides an immediate strengthening effect, the presence of microcracks under cyclic loading are known to substantially reduce fatigue strength and potentially the clinical lifetime of the ceramic restoration [74,75].

Microstructural variations within different commercial zirconia ceramic formulations are likely to significantly affect the extent and depth of phase transformation at the ceramic surface (known as the "transformed zone depth") following grit-blasting. That, coupled with extensive variability in surface grit-blasting protocols provides a complex recipe for optimization of mechanical properties and longevity at the resin-ceramic interface. Although there are vast amounts of laboratory evidence for improved interfacial adhesion to zirconia, little clinical evidence of durable bonding using rather complex and time-consuming procedures currently exists, and that which does, suggests that moderate-pressure grit-blasting and appropriate (phosphate-based) resin cements provide good clinical longevity [76].

Modern techniques for bonding to dental zirconia ceramics often include zirconate-coupling agents, which contain phosphate groups that readily bind to metal oxides and a photopolymerizable organic group with methacrylate functionality that provides immediate adhesive stability to the resin cement. Zirconia dental restorations with cements that include phosphate-based monomers such as 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) are strongly recommended due to a recognized

superior clinical durability [77.78], evidenced by long-term hydrolytically stable adhesion between the phosphate moieties of the cement and surface hydroxyl groups of the ceramic [61]. Even if the potential for increasing surface roughness is somewhat limited by the high hardness of zirconia, grit-blasting remains a critical step, not only to increase roughness for micromechanical retention, but also to increase the number of hydroxyl groups available at the surface [79,80].

The extent to which a zirconia ceramic surface should be roughened remains controversial. As previously mentioned, although grit-blasting has been reported to provide enhanced strength characteristics [81-84], others have suggested a strength-limiting effect [74,85]. A recent review article presented strong clinical evidence for long-term durability of zirconia ceramic restorations using grit-blasting with moderate air-pressure and phosphate-based cement systems [76]. The same review also quoted a previous clinical study, which identified the risk associated with the use of resin cements on unprepared (non grit-blasted) zirconia restorations. A failure rate of 13.3% over 53 months was reported [86], which would otherwise support pre-cementation grit-blasting of the ceramic surface. Even assessment of modern self-adhesive or "universal" resin cements still advocates mechanical modification of the ceramic surface by grit-blasting rather than relying on the self-adhesive quality of the cement alone [79,87].

Tribochemical modification is a popular technique to improve adhesive potential using airborne particle abrasion techniques. These methods typically employ silica-modified alumina particles in order to silicatize the ceramic surface to allow subsequent adhesion of a silane molecule [61]. Most in vitro studies report improved bond strengths to modified, silica-coated surfaces [66,83–89], although some show no significant difference with or without tribochemical treatment using the same resin cement [90], decreased strengthening effects [91,92] or even reported evidence of ineffective penetration of the silica-coated layer [93].

Born from the catastrophic failures of zirconia hip joint arthroplasties in the early 2000s, concerns exist over the effects of particle abrasion (whether that be from tribochemical or surface roughening treatments) on damage accumulation and low-temperature degradation of dental zirconia ceramics that may significantly affect their long-term clinical performance [94-96]. In order to eliminate the use of airborne particle abrasion techniques in an attempt to reduce the potential of long-term degradation, other (additive) surface treatments have been suggested such as a simple feldspathic surface glaze [97-100], which provides a surface layer that is amenable to etching and subsequently infiltrated with the resin cement system. A further technique known as, 'selective infiltration etching' [101] introduces a glass melt at relatively low temperature (750°C), which effectively thermally etches the surface providing micro-retentive features (without mechanical stress) for infiltration of the cement system. The authors report superior aged bond strength compared with grit-blasting. Other alternative surface modification techniques include fluorination pre-treatments [102], vapor deposition of silica-like "seed-layers" [103] and in situ silica nanoparticle deposition [104].

2.3 Adhesion test methods for the resin cement-ceramic interface

As with bond strength testing for resin adhesives and enamel/dentin (Section 1), interrogation of the mechanical properties and bond strength at the resin-ceramic interface is fraught with problems. The wide variation in strength data has been discussed previously, but generally relates to in vitro tests that barely relate to the clinical situation, inappropriate test method designs that do not effectively test the adhesive joint and other variables that are commonly not controlled.

The test methods in current use for assessing the bond strength of resin cement to glassy and polycrystalline dental ceramics are very similar to those used for the material-tooth interface described previously. Generally, macro-shear and tensile tests are most popular for adhesion testing of the resin and glassy ceramic interface, although macro-shear remains the predominant testing modality for

studying the adhesion of resin cements to zirconia [105,106], likely due to ease of specimen preparation without the need for cutting fully sintered zirconia into rectangular- or hourglass-shaped specimens. However, shear tests have long been criticized for their inability to accurately forecast clinical outcomes [16,107,108], as well as for unpredictable stress distributions which lead to failure initiation away from the adhesive interface with the inevitable misinterpretation of the meaning of the bond strength data [19]. Although shear-bond strength tests are the most used for resin cement-zirconia adhesion, they are reported as providing generally the lowest bond strength values and are the least discriminative compared with micro-shear, macro- and micro-tensile tests [106].

Alternative, less common, approaches include tensile bond strength by flexural testing [100,109,110,]. Here, the bonded surface is placed centrally under three- or four-point loading, the latter method requiring less accuracy in alignment of the adhesive joint directly beneath the central load (Figure 4). Nonetheless, for either method, obtaining a pure state of tension through the adhesive interface is difficult. The perceived advantages of interrogating the adhesive interface using a four-point flexure are ease of specimen preparation and better fixation and alignment over direct tension tests. Polycrystalline ceramic blocks can be machined in their pre-sintered state into over-sized bar-shaped specimens that allow for shrinkage following full densification (Figure 4). The test configuration should allow a span width to specimen depth ratio of greater than 10 in order to prevent shear stresses within the adhesive joint [100,109]. Previous researchers have used specimen dimensions of 25mm length, 2mm width, 2mm depth [109] or 5mm width according to ASTM C164-11 [100,111]. It has been suggested that the maximum tensile load at the convex surface of the four-point bend specimen and subsequent gradient stress field along the load axis is more clinically relevant than either direct tension or shear tests [100]. More recently, researchers have utilized a wedge-loaded double-cantilever-beam

testing method to accurately measure the interfacial fracture resistance of adhesively bonded restorative materials since such a testing configuration produces a pure Mode I fracture [112].

Regardless of the testing method and their perceived advantages and drawbacks, fractographic analysis under high magnification is essential to better understand the characteristics of failure. The "adhesion zone", that is the "region in which the adhesive interacts with the two substrates [resin composite and ceramic] to promote bonding" should be fully interrogated in order to identify the mode of failure, which allows for a complete description of the failure process without relying on bond strength data alone [113].

3. The Core and Veneering Ceramic Interface

A definition of interface in dentistry is not uniquely assigned to adhesive dentistry but also includes sintered and soldered joints. Those interfaces are commonly found in prosthetic dentistry and mainly represent the application of veneers to metal or ceramic core substructures in fixed partial dentures (FPDs), the purpose of which is to enhance the overall esthetic quality of the final prosthesis. Especially, issues related to metal-ceramic and ceramic-ceramic interfaces are repeatedly addressed and updated in dental research.

In dentistry, the ceramic veneering layer is applied in largely varying thicknesses and curvatures that are unique to each single FPD (Figure 5). Veneers thereby serve many purposes. First of all, the esthetic appearance of glassy feldspathic ceramics is very natural and second, with the use of fine-grained, reinforced glass-ceramics, the hardness and thus the potential abrasivity of a restoration surface has been adjusted to better mimic that of the natural, human enamel [114].

3.1 Internal residual stresses

Dental veneering in the majority of cases is a sintering process based on a slurry technique, commonly conducted in repeated cycles by building up layer-by-layer. In recent years however, the method of soldering or even cementing CAD/CAM veneers onto a framework substrate has become relatively popular [115]. This alternative manufacturing protocol intended to minimize internal stresses at the core-veneer interface. This development was intrinsically related to the rapid improvement in CAD/CAM technology, which provided a perfect fit between both layers.

The key requirement for sintering veneers onto either metal or ceramic substrates is an optimal, mechanically stable adhesive joint [116,117]. Several factors contribute to this adhesion. Chemical forces are mainly responsible, though they are assisted by microretentive interlocking via surface

roughness of the substrate [118,119]. Metal alloys suitable for the veneering process contain certain metals, which preferably oxidize at the interface during sintering (such as iron, chrome, tin, indium, etc.) and thus form a primary chemical bonding to the silica of the veneering ceramics. On the other hand, a (micro-) rough substrate surface, achieved by e.g. sandblasting techniques, is known to increase the interfacial adhesion [119-121]. On a macroscopic level, the contraction forces that build-up during cooling from sintering temperatures is reported to further increase the interfacial adhesion [122-127]. A common ceramic slurry shrinks between 20 – 30% during sintering. Part of the contraction occurs due to densification as the liquid phase is removed and the particles begin to fuse. In addition, there is a solid-state contraction defined by the thermal coefficient of expansion (CTE) as the material cools below its glass transition temperature Tg [124,128].

The shrinkage behavior of the veneer and the core materials would ideally be slightly mismatched in order to produce a build-up of internal stresses at the surface [129-131]. The CTE of the veneering material has traditionally been selected to be slightly lower compared to the substrate CTE (within a range of maximum 10% mismatch) [128]. Figure 6 shows the result of the calculation of expected stresses in a 3 mm thick veneer layer due to a CTE mismatch of 10%. The theory explains this by the introduction of reinforcing, compressive stresses in the weak and brittle veneering ceramic [123-125,132]. However, individual dental restorations show a large variation in veneering thickness and curvature. Alternating concave and convex curvatures combined with e.g. thick cusps and thin fissures accounts for a multiaxial stress state in the veneering ceramic [125,133]. Beneficial compressive stresses thus need to be compensated by deleterious tensile or shear stresses [132]. As a consequence, the current technical advice for the veneer CTE should be to match the substrate CTE as close as possible in order to minimize internal residual stresses [134,135]. Further, slow cooling of the core-veneer construct should be adopted following firing to prevent excessive thermal gradients and deleterious interfacial

transient stresses, which may otherwise result in delamination, chipping and/or fracture of crowns in service [135].

3.2 The zirconia-veneer interface

The adhesion between feldspathic veneers and zirconia, however, is not completely understood. Early research on the zirconia interface to borosilicate glasses proved a certain solubility of zirconia in the glass at elevated temperatures of 815 or 1200°C [136]. It has also been shown that zirconia enters the glass network as six- or eight-coordinated network modifiers [137]. Durand et al. [138] analyzed the interface between veneer and zirconia using Raman spectroscopy, and detected an interdiffusion zone of about 2 µm [138]. Other work has shown that zirconia act as nucleating elements for glass crystallization and is able to enhance crystal growth [139]. Figure 7 shows a cross section of the interface under TEM. No voids, thus complete wetting and sealing has been shown, even on rather rough sandblasted zirconia surfaces [140,141]. However, some disintegrated zirconia particles become solved in the glass matrix, most likely due to the sandblasting process. Tholey et al. [141] further suggested that tetragonal to monoclinic phase transformation could be triggered by the veneering process via local low temperature degradation induced by the veneering liquid.

3.3 Mechanical properties of bilayer interfaces

The quantification of the interfacial adhesion and assessment of internal stresses is of central interest to the practical use of veneered restorations. Several techniques and protocols have been developed in the past in order to measure the bonding potential. Mainly of an indirect nature, the procedures descriptively monitor and test crack initiation, either due to mechanical or dynamic thermal loading. The most common tests reported in literature are based on so-called “crunch-the-crown”

principles [131]. Those tests, however, due to the complexity of the veneered restorations, are not very sensitive and discriminatory, and provide only little information upon the underlying principles and mechanisms [142]. Further, the load-to-failure data from those tests far exceeds the maximum masticatory forces in vivo and thus is lacking clinical relevance. More standardized research has been developed into (bi-) axial strength testing of veneered and flat specimens [124]. Based on these tests, variations in layer thickness and orientation, crack initiation and crack path development can be investigated. The most prevalent procedure, however, has been adopted as a standardized procedure in ISO 9693 [143] and ISO/DIS 9693 [144]. The test is originally developed to determine the interfacial resistance of metal-ceramic bilayers against crack initiation or debonding in three-point flexure [119,145]. Basically, the test specimens are made of a thin substrate and a short veneer layer, which is loaded under bending conditions until initial cracks are formed at the interface between both layers. The veneered side of the specimens is thereby placed under tension. The procedure will soon be extended to ceramic-ceramic systems (ISO/DIS 9693-2) and also will include a test procedure for dynamic and cyclic thermal shock stress testing in order to prove the thermal compatibility of material combinations.

The ISO procedure is designed firstly for practical use, e.g. is easy to perform by dental technicians (to control the veneering procedure), but provides only little insight into the underlying mechanisms. Because it is conducted on flat specimens, the interpretation of the data and its relevance to the clinical situation is limited. For example, the orientation of the veneer layer on the tensile side of the bending setup does not realistically reflect the clinical scenario. Interestingly, ISO 9693-1 provides a numerical solution for interfacial shear strength calculations. This approach uses the elastic modulus, thickness and failure load as input variables, but only of the substrate, not of the veneering material. Further, the effect of mismatch in CTE remains uncertain. Recently, however, Schneider and Swain [121] applied a linear elastic approach to the ISO test setup by calculating strain energies and proposed to extend the ISO procedure by the calculation of an internal fracture toughness parameter.

3.4 Clinical Findings on Veneered Zirconia Restorations

In recent years, with the success of zirconia as a framework material, the clinical experience revealed some serious concerns related to the veneering procedure. In contrast with restorations on metal substrates, the chipping rate for zirconia-veneered restorations was found in early studies to be significantly increased. Failure rates up to 25% after 2.5 years [146], 36% in 5 years [147], or 32% after a period of 10 years [148] were reported, with more severe clinical consequences often leading to a complete restoration replacement [149-151]. Figure 8 shows an example of a zirconia veneered 5-unit bridge with a severe chipping of the buccal wall of the most distal abutment tooth. However, more recent clinical reports on the success of veneered zirconia restorations have shown a more promising performance compared with the earlier studies and a comparable success rate as with metal based FPDs [153,154].

3.5 Measurement of Residual Stresses

Inspired by the chipping issue, intensive research has developed to acquire more insight into the stress state of a sintered all-ceramic core-veneer interface. Pioneering work has been published by Swain [132], in which he first discussed the specific features of Y-TZP and identified how its low thermal diffusivity ($D= 0.74 \times 10^{-6}$ m^2s^{-1}) compared to metals ($D= 1.1 \times 10^{-4}$ m^2s^{-1}) significantly contributed to the build-up of internal residual stresses within the veneer layer. The influence of a low thermal diffusivity is of practical importance during cooling a restoration from the veneering temperature [124, 134, 135, 155-157]. Fast cooling is thereby identified as a major contributor to the internal stress build-up and has been implicated as the reason for the increased chipping rates on

zirconia [123, 132,158]. As a consequence, current practical recommendations to dental technicians include a slow cooling protocol in the final step of the veneering procedure [123, 132,159]. The dental industry has adapted this recommendation to their sintering furnaces, which today include slow cooling programs or retarded door opening times.

Consequently, the role of residual stresses in bilayer structures and their measurement has been intensively addressed in dental research. The majority of experiments in the past have focused on the measurement of dimensional changes in the veneer layer due to variation of processing variables such as veneer thickness, substrate curvature, firing conditions, CTE mismatch, etc. [127,129,160-163]. More recently, it has become possible to investigate and quantify the amount of residual stresses in clinically relevant model structures and to relate them to their effect on bilayer integrity and strength [135]. The precision of these techniques strongly relates to the resolution of the deformation measurement. Optical and profilometric techniques, as well as surface evaluation via strain gauges, have been investigated. The method of hole-drilling into the veneer layer perpendicular to the core-veneer interface also provides an opportunity to measure the dimensional changes within the veneer layer [164]. The drilling procedure induces a release of local stresses and thus generates a micro-relaxation in the veneer layer depending on the residual stress state. Circular strain gauges attached to the surface sensitively measure the dimensional changes. However, problems arise due to an extreme local heating during drilling, which might significantly change the local stress state, and even result in micro cracking [165]. Further, this approach provides only global surface information on residual stresses in a plane-strain configuration parallel to the interface. The method consequently involves an invasive removal of incremental surface layers.

Another interesting approach focuses on light transmittance of polarized light through thin, transparent or translucent slices utilizing the birefringence effect [134,159]. Double refraction (birefringence) is measured based on the photoelasticity of materials and results from the retardation of

polarized light through a translucent material with heterogeneous density. Stress-free glasses and ceramics, which are optically isotropic, become anisotropic when exposed to internal residual stresses or external mechanical actions [166,167]. Figure 9 shows an example of the photoelastic effect in thin crown slices, where a two-dimensional mapping of stress distribution in tension and compression becomes possible. The advantage is the ability to investigate the stress state in different planes, e.g. perpendicular to the interface. Still the method presents an invasive approach due to the cutting process and an expected strain release, thus requiring a careful, slow cutting procedure under continuous water-cooling.

In order to better understand crack development and crack path profiles, indentation crack measurements present an interesting complement to the aforementioned techniques [168-174]. Basically, a surface indentation (e.g. using Vickers indenters) initiates and drives cracks into a plain ceramic surface thereby allowing analysis of the length, nature and orientation of the respective crack paths [175]. Crack deviations close to the interface, especially in a residual stress field, became a matter of interest [176]. Indenter modifications using Knoop or Berkovich indenters allow for targeted orientation of the crack initiation process [175]. Indentation analysis can be easily performed on individual plane orientations [177]. With the use of nanoindentation techniques, this kind of analysis can be performed close to the core-veneer interface.

Residual stresses in a surface or sub-surface layer can also be determined using X-ray diffraction [178]. The microstructural lattice space of a crystalline material changes due to internal residual stresses. Based on Bragg's law, X-ray diffraction presents a precise measure of the lattice parameters in order to quantitatively describe a residual stress state via lattice strain measurements [179]. This technique provides high resolution and precise outcomes. The penetration depth of X-rays, however, is limited to a few micrometers from the surface and thus involves an invasive removal of incremental surface layers.

An essential tool for analyzing the residual stress state in bilayer structures and complementary to all experimental approaches is the numeric approximation of the problem using finite element (FE) models [156,163,180-183]. The major advantage of this tool is the simulation of dynamic thermal or mechanical processes as they happen during sintering or intraoral mastication [163,182]. Also unique to this method is its application to individual geometries, allowing the research findings on planar structures to potentially be extrapolated to curved and thus more clinically realistic geometries [181, 184, 185]. Common difficulties with FE modeling is that the significance is extremely limited to step-by-step verification and confirmation of the model with experimental results.

3.6 Future Perspectives

The recent developments from industry have aimed to overcome the problems regarding internal residual stress build-up. The idea behind the new approaches involves a simultaneous CAD/CAM manufacturing of both parts followed by a separate bonding step [115]. Two different concepts are currently marketed. The “CAD-On” technique (Ivoclar Vivadent, Schaan, Liechtenstein) includes a heating procedure and soldering an e.max CAD veneer via a fusion glass-ceramic onto a zirconia framework [186] while the “Rapid Layering Technique” (Vita Zahnfabrik, Bad Säckingen, Germany) favors an adhesive cementation of both layers. However, to date, little is known about the clinical success, manufacturing quality, process complications and basic research for the two concepts. Figure 10 shows a soldered interface between an e.max veneer and zirconia framework foretelling some of the possible complications and deficiencies of those techniques. The image is taken from a clinically failed 3-unit bridge after 18 month in situ.

Conclusions

A variety of materials and material combinations are used for the repair or replacement of oral and craniofacial tissues, resulting in numerous types of material interfaces and the quality of the interface is very much dependent upon the individual properties, as well as the many factors that affect the adhesion between the two. The interfacial properties of either material-tooth, or material-material adhesive joints and use of accurate and relevant testing methods are essential components for understanding the behavior and clinical performance of the final construct.

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Figure captions

Figure 1: Microstructure of enamel (left) and dentin (right) revealed by acid treatment showing the dense structure of enamel, which is composed of rods of hydroxyapatite mineral, and the porous structure of dentin, which is composed of hydroxyapatite mineral with protein surrounding the tubules.

Figure 2: Microstructure of the enamel-dentin junction.

Figure 3: Microstructure of the interface between a resin adhesive and dentin treated with a strong acid showing a defined hybrid layer (HL) of a few micrometer thickness (left) and a milder acid in which the hybrid layer is very thin (right).

Figure 4: The adhesive joint between resin cements can be investigated using four-point flexural strength testing. Over-sized polycrystalline ceramic bar-shaped specimens are cut from pre-sintered blocks (a) to allow for full densification shrinkage prior to testing. The adhesive surface is prepared (grit-blasted, silica-coated, surface glazed, etc.) (b) and re-joined using a suitable resin cement material. The interface is positioned centrally under the load within a four-point bend apparatus and tested under flexure with failure assumed to initiate through the adhesive under tensile stress (c). To reduce the effect of shear deformation under flexure a span width to specimen depth ratio of at least 10 should be used [100, 109].

Figure 5: Cross section through a veneered all-ceramic molar restoration showing the varying thickness and curvature of the veneer layer.

Figure 6: Calculation of internal stresses in the core and veneer layers as a result of the calculation of expected stresses in a 3 mm thick veneer layer due to a coefficient of thermal expansion mismatch of 10%

Figure 7: TEM cross section of a sandblasted zirconia-veneer interface showing perfect wetting and interlocking (arrows) [140] (Grigore 2013).

Figure 8: Veneer chipping of a zirconia veneered 5-unit bridge after 2.6 years in situ [152].

Figure 9: Residual stress map of a veneered cross section under polarized light (birefringence), highlighting the development of internal stresses upon different cooling protocols [159]

Figure 10: SEM image of a veneer soldered onto a zirconia framework.

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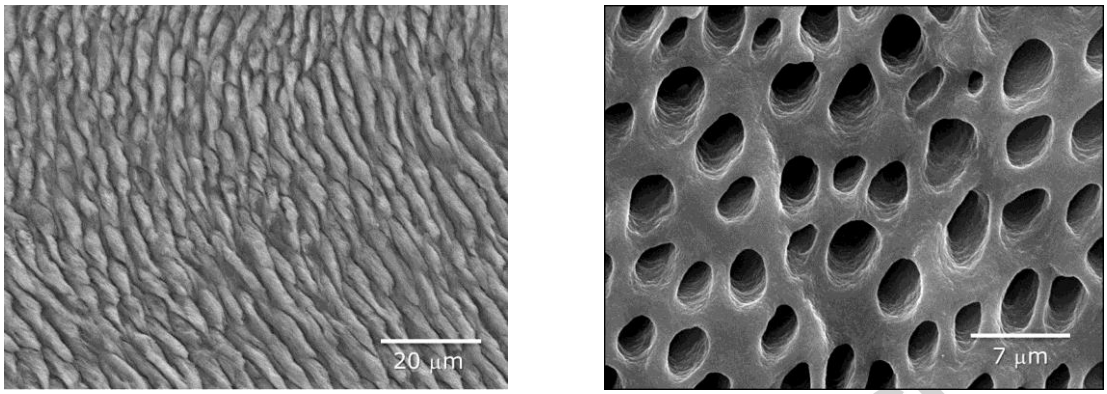

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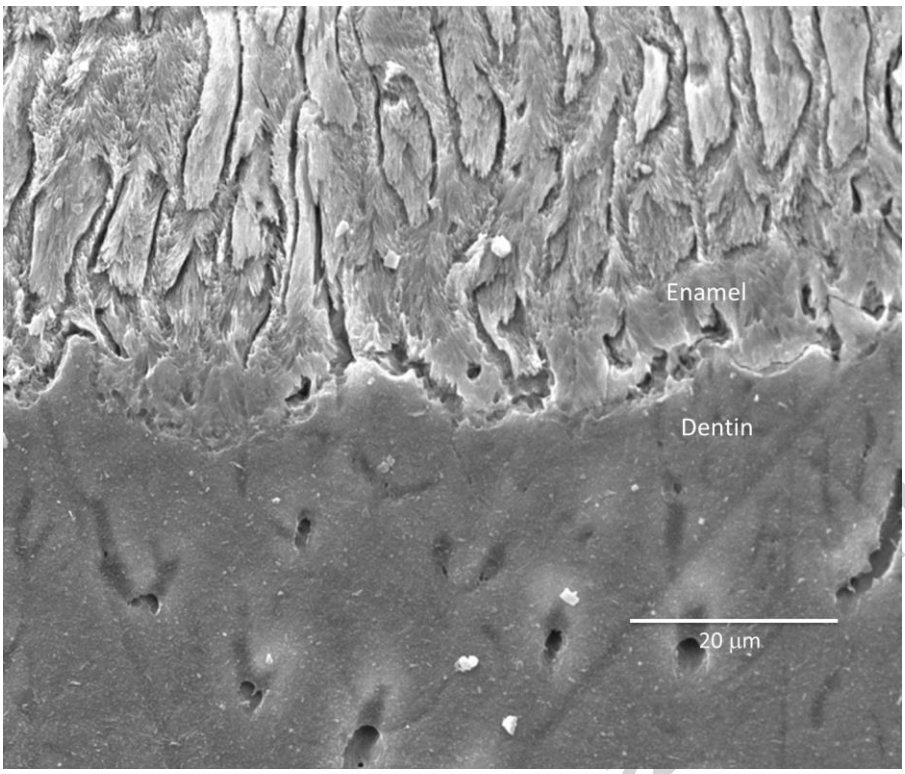

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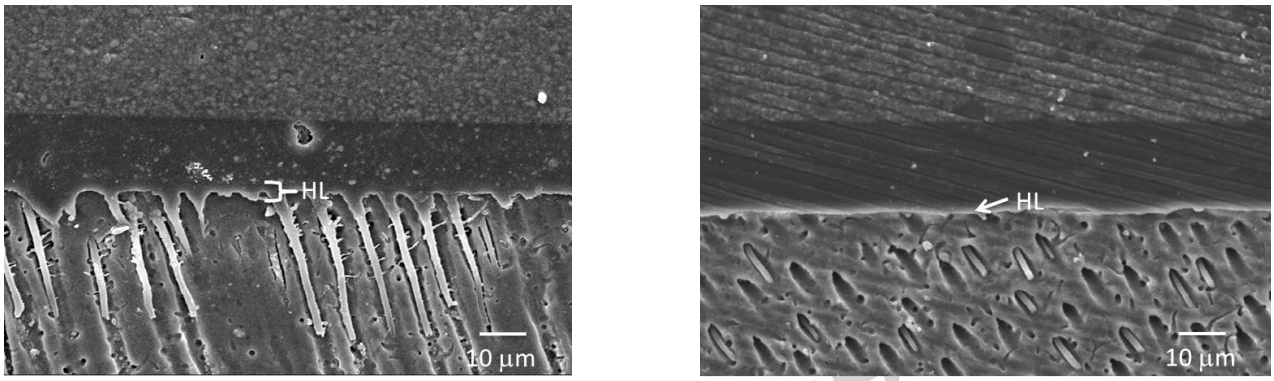

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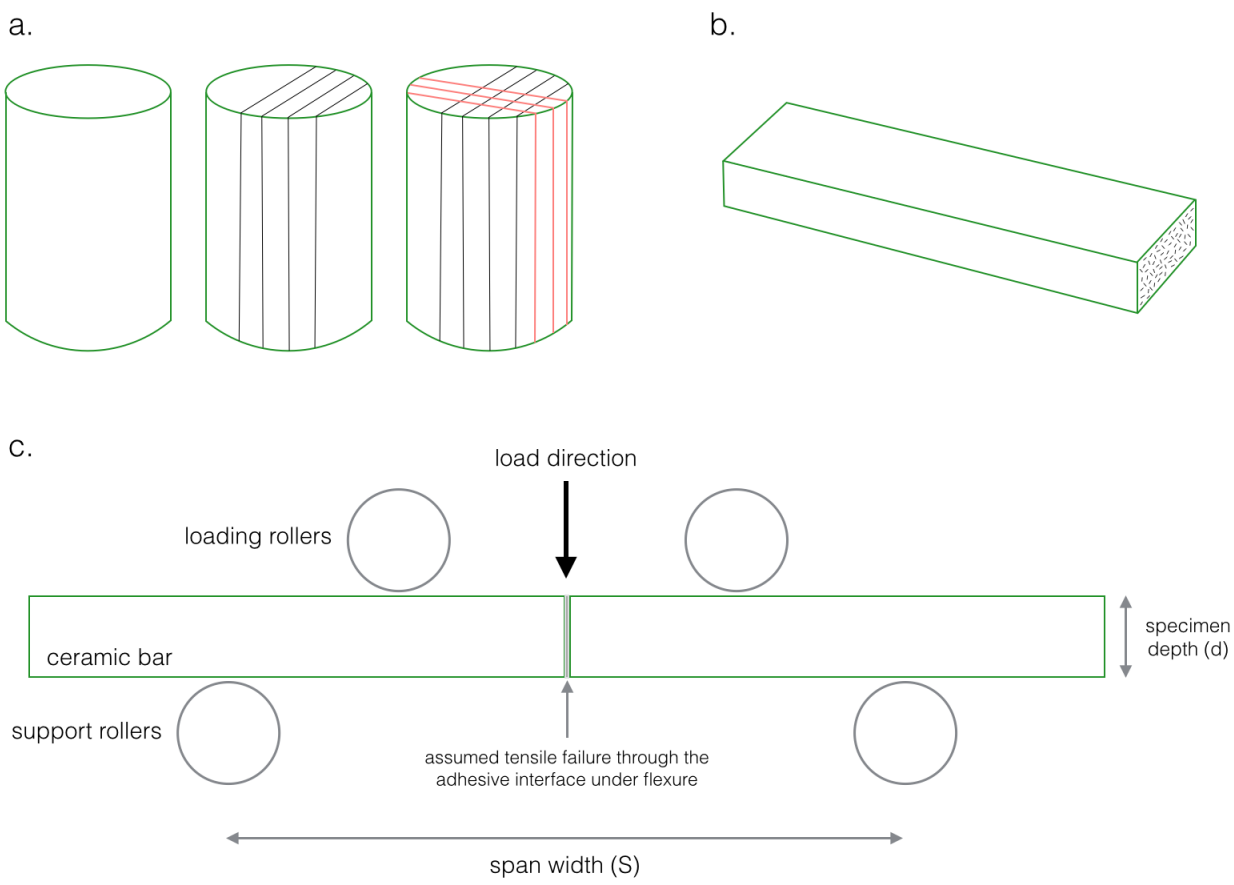

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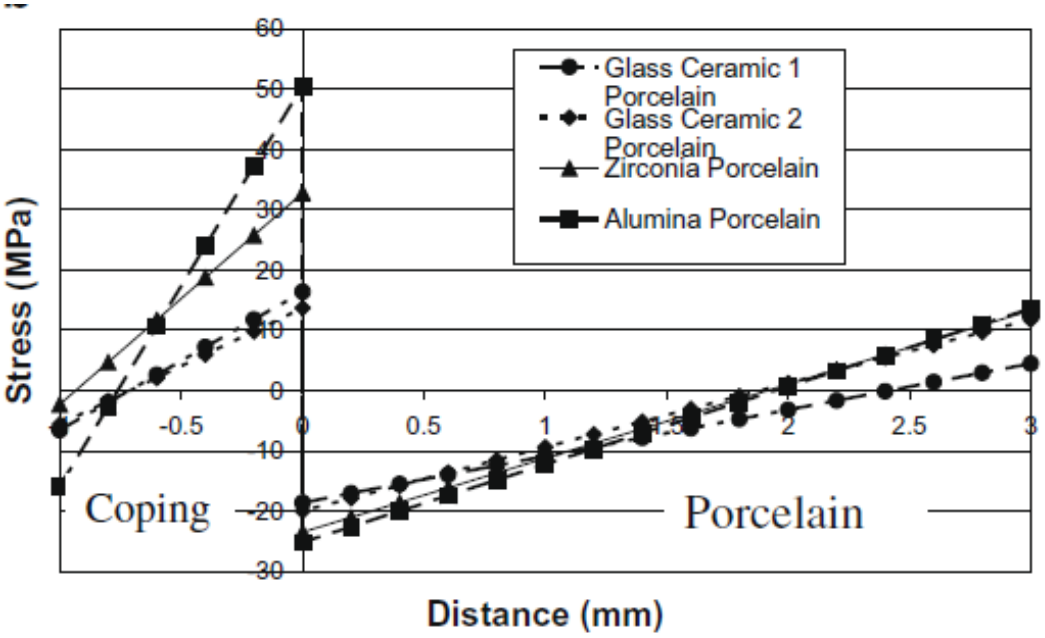

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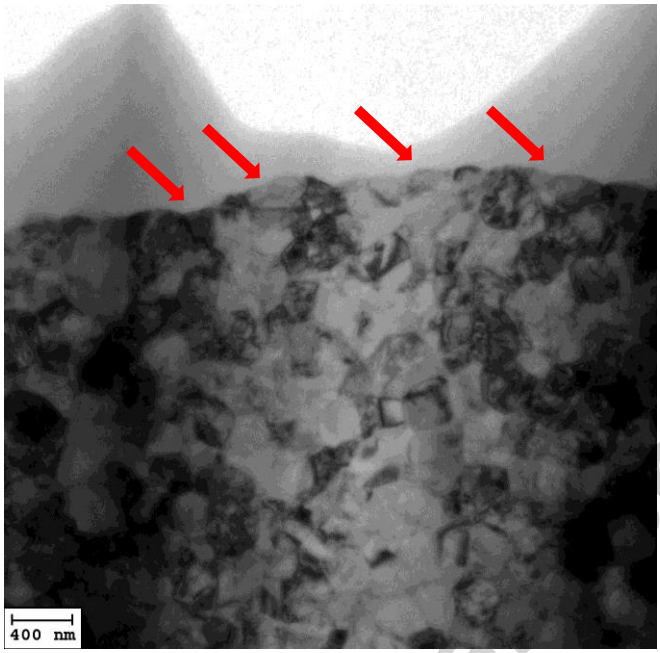

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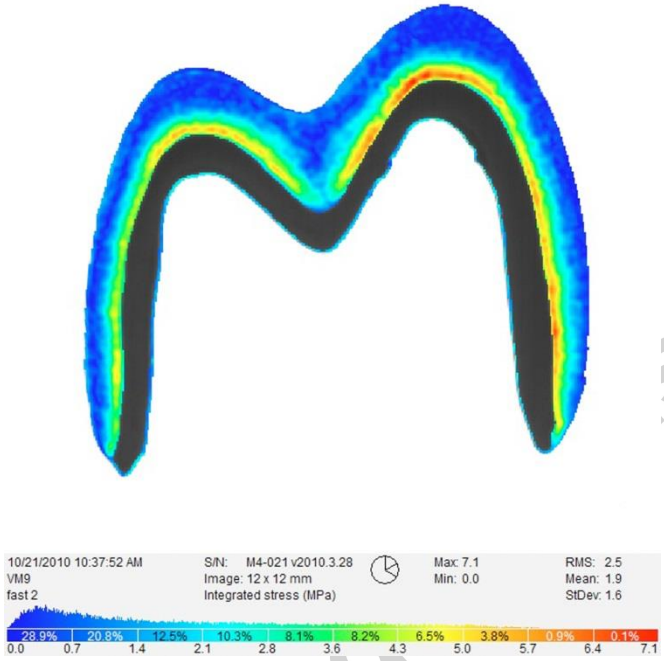

Figure 10: SEM image of a veneer soldered onto a zirconia framework.